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THE IGNITION OF PRECIPITATES WITHOUT THE USE OF THE BLAST LAMP.

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The introduction of compressed air in general laboratory equipment has caused chemists to heat precipitates over the blast lamp to a greater extent than when the foot bellows was in general use. Much of this blasting is not only unnecessary but instead of increasing accuracy it tends to give inaccurate results, for not only is there greater danger of mechanical loss of the precipitate but, as has been shown by the Committee on Platinum Laboratory Utensils of the American Chemical Society,[1] even the best platinum loses weight in appreciable amounts at a temperature in the neighborhood of 1,200° C. This temperature can be obtained in a crucible with some blast lamps, while with others of apparently the same construction such high temperatures are not given..

It is almost a universal custom to blast lime, alumina, and silica to constant weight. That blasting is unnecessary for lime has been shown by Hillebrand,[2] and his conclusions that the heat of a Bunsen burner is sufficient for lime have been confirmed in this laboratory. He, however, prescribes blasting both silica and alumina, and with ordinary short periods of heating over a Bunsen burner, his conclusions as to the necessity of blasting are correct. Some preliminary experiments made on precipitated silica and aluminum hydrate, however, indicated that two or three hours at the highest temperature ordinarily obtainable with a good Bunsen burner were sufficient to reduce these substances to constant weight and it was deemed desirable to ascertain the necessary conditions for igniting them.

All of the work described in this paper was done using the Washington city gas, which is mainly an enriched water gas mixed with some true coal gas. Various temperature measurements were made by inserting a thermo element through a hole in a platinum crucible cover, and it was found that at a point about 1 mm from the bottom of a 15 cc platinum crucible the ordinary types of Bunsen burners gave temperatures in the neighborhood of 950° C. Measurements were made, using old-style Bunsen burners (where the air supply is controlled by a collar and two holes at the base of the chimney) and a

[1] J. Ind. Eng. Chem., 1911, *3:* 686-691. [2] U. S. Dept. Int., Geological Survey Bul. 422, p. 119.

number of more modern types, having improved methods of controlling the air and gas supply. To the surprise of the experimenters there was practically no difference in temperature when different types of burners which are in common use were employed, the old-style Bunsen giving about the same temperature as modern types when of the same size. Similar experiments were made with Meker burners, and it was found that both large and medium Meker burners gave the same temperatures in the crucible; that is, about 1,025° C., or about 75° higher than that obtained by an ordinary burner. The small size Meker burner gave a lower temperature than the ordinary burner, but it was evident that the flame from this small burner was not large enough to properly heat the size crucible used, and it would probably be equally efficient for a smaller crucible.

Under similar conditions blast lamps gave temperatures varying very much more than the burners. Using different blast lamps, temperatures of from 1,075° to 1,260° C. in crucibles were obtained. Measurements of the flame temperatures by putting exposed thermo elements in the hottest part of the flames gave temperatures of from 1,250° to 1,290° C. for the ordinary type of burners, 1,300° to 1,320° C. for Meker burners, and 1,410° to 1,580° C. for blast lamps. The gas consumption per hour and the approximate temperature, 1 mm above the bottom of a covered 15 cc crucible, of the burners and blast lamp used in these tests were as follows:

Gas consumption and temperatures of three flames.

Flames.	Gas consumption per hour.	Approximate temperature 1 mm above bottom of covered crucible.
	Cubic feet.	° *C.*
Bunsen burner (E. & A. Universal)	7.25	950
Meker burner (medium size)	5.13	1,025
Blast lamp	18.75	1,150

EXPERIMENTS WITH SILICA.

In order to eliminate as far as possible all errors except those due to heating, a quantity of silica was prepared by decomposing sodium silicate with hydrochloric acid, evaporating to dryness, digesting with hydrochloric acid, and washing by decantation. This material was heated at a temperature of 105° C. until it was apparently dry, but no attempt was made to dry to constant weight. Different portions of this powder were heated in various ways. Table 1 gives the results of twelve experiments which were conducted by heating the silica at the highest temperature obtainable with burners used, for 3 hours before the first weighing and then for 1 hour more over a burner, after which it was weighed and heated for 15 minutes over a blast lamp.

TABLE 1.—*Results of experiments with silica.*

(Heated 3 hours, then 1 hour, followed by 15 minutes blasting.)

Experiment No.	Weight of material taken.	Weight—						Change in weight.	
		After 3 hours over burner.		After 4 hours over burner.		After 15 minutes blast, following 4 hours over burner.		During fourth hour of heating.	Due to blast.
	Grams.	*Grams.*	*Percent.*	*Grams.*	*Per cent.*	*Grams.*	*Per cent.*	*Mg.*	*Mg.*
1	0.8774	0.7902	90.06	0.7828	89.22	0.7827	89.21	−7.4	−0.1
2	.7749	.6925	89.36	.6923	89.34	.6919	89.30	− .2	− .4
3	1.0356	.9286	89.70	.9284	89.68	.9284	89.68	− .2	.0
4	1.0762	.9573	89.95	.9571	89.93	.9571	89.93	− .2	.0
5	1.3341	1.1972	89.74	1.1972	89.74	1.1967	89.70	.0	− .5
6	1.2378	1.0878	87.88	1.0878	87.88	1.0878	87.88	.0	.0
7	1.0592	.9581	90.46	.9577	90.42	.9577	90.42	− .4	.0
8	.9914	.8976	90.53	.8973	90.51	.8971	90.49	− .3	− .2
13	.6939	.6284	90.56	.6283	90.55	.6283	90.55	− .1	.0
14	.7888	.7124	90.32	.7125	90.33	.7124	90.32	+ .1	− .1
15	.9178	.8309	90.63	.8302	90.55	.8301	90.54	− .7	− .1
16	1.0184	.9214	90.47	.9206	90.39	.9207	90.40	− .6	+ .1

TABLE 2.—*Results of experiments with silica.*

(Heated to redness, then blasted.)

Experiment No.	Weight of material taken.	Weight—						Change in weight.	
		After 15 minutes blasting.		After 20 minutes blasting.		After 25 minutes blasting.		Between 15 and 20 minutes blasting.	Between 20 and 25 minutes blasting.
	Grams.	*Grams.*	*Per cent.*	*Grams.*	*Per cent.*	*Grams.*	*Per cent.*	*Mg.*	*Mg.*
9	1.1373	1.0307	90.61	1.0300	90.56	1.0300	90.56	−0.7	0.0
10	1.1638	1.0622	91.26	1.0616	91.22	1.0615	91.21	− .6	− .1
11	.9352	.8473	90.60	.8457	90.43	.8443	90.28	−1.6	−1.3
12	1.0174	.9180	90.23	.9179	90.22	.....	.....	− .1	.....

In all of the experiments in this work a clay cylinder about 4 inches in diameter was set around the crucible. This radiator does not appear to raise the temperature at the bottom of the crucible but does probably give a more uniform temperature over the whole of the inside. Table 2 shows the results of four experiments conducted in the ordinary way; that is, the crucibles were heated to redness and then heated with the blast lamp for 15 minutes before first weighing. They were then heated for five minutes, weighed, and heated for another period of five minutes and weighed. In the odd-numbered experiments (1, 3, 5, etc.) the burner used was of ordinary type (E. & A. Universal); in the even-numbered (2, 4, 6, etc.), the medium-sized Meker burner was used.

Experiments 1 to 6, inclusive, in Table 1, were heated rapidly up to the maximum temperature that could be obtained with the burner used. The remaining experiments were heated up gently at the beginning, taking about 15 minutes to bring them up to the maximum

temperature, and then held at this temperature for the periods indicated in the table. In all of the tests recorded in Tables 1 and 2 the crucibles used were cleaned with sand between the experiments and weighed only at the beginning, the calculations being based upon that weight. The experiments were conducted in pairs, and a control crucible was heated for about three minutes to about the temperature of the crucibles containing the precipitates, placed in the desiccator with them, and weighed immediately after weighing them. The control crucible was cleaned only once during the series. Before this cleaning its weights varied 0.6 mg, the lowest weight being 0.2 mg below and the highest weight 0.4 mg above the initial weight. These changes were evidently not due to loss of weight in the control, since the highest weight recorded was next to the last before cleaning, and the last weight was 0.2 mg greater than the first weight. After the cleaning the maximum variation of this control crucible was 0.3 mg.

As would naturally be expected, the material used is not perfectly homogeneous and the percentages of silica found for the various periods of heating can be used only as pointing to probable mechanical loss of part of the silica. Experiment 1, for example, shows a loss of 7.4 mg during the fourth hour of heating, but the low percentage indicates that some silica was lost (blown out of the crucible). Experiment 5 shows a very much lower percentage than the rest of the series, but as the first six experiments were all heated up rapidly at the beginning it is probable that some material was lost here also. It will be noted that in the experiments recorded in Table 1, in no case except in the first experiment was there a loss of as much as a milligram after the first heating. The small changes in weight are no greater than apparent changes in weight of the same object at different times.

The results in Table 2 indicate that it is more difficult to get constant weight by heating to the temperature of a blast lamp as soon as the material has been heated up to bright redness than it is if all the heating is done at a lower temperature and for a longer time. The results in Table 1 indicated that nothing volatile was driven off after three hours heating over the burner, but as no attempt had been made to check possible changes in weight of the crucible during the individual experiments another series of tests was run later. The results of this series are recorded in Table 3. A portion of the same lot of silica was used. The material had been kept in a screw-top jar with no particular precautions to guard against slight changes in the moisture content, so that the percentages in the results differ considerably from the earlier experiments. Since the results in Table 1 indicated that silica was reduced to constant weight in three hours heating, in most of the experiments recorded in Table 3 weighings were made at periods of one, two, and three hours and finally after 15 minutes

over the blast lamp. After each experiment the crucible used was wiped out and weighed. As in the first series a control crucible was heated for about three minutes to about the temperature of the crucibles containing the silica, placed in the same desiccator, and weighed immediately after them. Twenty weighings of this control crucible were made. The extreme variations in all of these weighings was 0.4 mg. An inspection of the weights of this crucible shows that the variations in weight were not due wholly to volatilization of the crucible, but were errors in weighing. Since the crucibles containing the silica were cleaned between the experiments the individual weighings are not given in the table but only the apparent change in weight from the beginning to the end.

TABLE 3.—*Results of experiments with silica, showing changes in weight of crucibles.*

Experiment No.	Weight of material taken.	Weight— After 1 hour over burner.		After 2 hours over burner.		After 3 hours over burner.		After 3 hours over burner and 15 minutes over blast.		Change in weight of crucible from beginning to end of experiment.	Weight of control. At beginning of experiment.	Weight of control. At end of experiment.
	Grams.	*Grams.*	*Per ct.*	*Grams.*	*Per ct.*	*Grams.*	*Per ct.*	*Grams.*	*Per ct.*	*Mg.*	*Mg.*	*Mg.*
17	1.1421	.....	.....	1.0147	88.85	1.0145	88.83	1.0147	88.85	−0.3	16.0353	16.0351
18	1.0293	.....	.....	.9126	88.66	.9123	88.63	.9122	88.62	− .6		
19	.9746	0.8661	88.87	.8653	88.78	.8650	88.75	.8651	88.76	.0	16.0350	16.0350
20	1.0235	.9096	88.87	.9086	88.77	.9085	88.76	.9083	88.74	− .6		
21	.9278	.8227	88.67	.8220	88.60	.8220	88.60	.8219	88.59	− .1	16.0353	16.0352
22	1.0891	.9654	88.64	.9644	88.55	.9642	88.53	.9642	88.53	− .7		
23	.8091	.7181	88.75	.7179	88.73	.7175	88.68	.7174	88.67	− .4	16.0350	16.0349
24	.9972	.8842	88.67	.8843	88.68	.8838	88.63	.8837	88.62	− .5		
25	.8684	.7706	88.74	.7700	88.66	.7697	88.64	.7695	88.62	− .4	16.0350	16.0349
26	.7749	.6597	88.56	.6593	88.52	.6590	88.49	.6590	88.49	− .6		

EXPERIMENTS WITH ALUMINA.

The preliminary experiments that were made with aluminum hydrate prepared in large quantities and ground after drying in an air bath showed that this material could be ignited to constant weight without the use of the blast, but as the dried aluminum hydrate was ground before igniting it was thought that constancy in weight might not be obtained as easily by starting with precipitates actually obtained in analysis. Therefore, 16 determinations of alumina in ammonium alum were made. Table 4 gives the summary of results of these experiments. All the reagents used contained less than 0.5 mg of nonvolatile residue in 100 cc. All precipitates and boilings were made in platinum dishes. The material taken was dissolved in 25 to 30 cc of water, 25 cc approximately five times normal hydrochloric acid added, the solution made barely alkaline by the addition of five times normal ammonia, and boiled. Experiments 1 and 2 were washed only with water. In the others the washing was with

faintly alkaline ammonium nitrate about one-fifth normal. In experiments 3 to 8, 11, 12, 15, and 16 macerated filter paper was added. After washing the precipitates, they were placed with the papers in crucibles, covered, and heated very gently, being held about 8 inches above the top of the flame until the papers began to smoke. The crucibles were then lowered to within about 5 inches of the flame and heated until all smoking had ceased and nearly all of the carbon was destroyed. They were then lowered until the flame just touched and heated the lower part to a dull red and held at this temperature until the carbon was burned off. The ignition to this point required from 45 to 60 minutes. Covered crucibles were then placed in the full heat of the burners for various periods, which are indicated in Table 4. Clay cylinders were placed around the crucibles as in the experiments with silica and the burners used were the same as used in those experiments.

The odd-numbered experiments (1, 3, 5, etc.) were heated with the ordinary type of burner (Universal) and the even-numbered experiments (2, 4, 6, etc.) were heated with medium-sized Meker burners. The corrections which were made for a number of these determinations are given in Table 4. They were made by evaporating the filtrate from the first precipitation in platinum, igniting to drive off the ammonia salts, dissolving the residue in hydrochloric acid, and precipitating the alumina by the addition of a small amount of ammonia. These correction precipitates were ignited in another platinum crucible. After ignition the pair of crucibles used for determinations and the control crucible which was heated for about three minutes to approximately the temperature of the others were placed in the same desiccator and weighed after being kept there for about 15 minutes. The errors due to weighing seemed to be of about the same magnitude as those in the silica determinations, the second weight of the control crucible varying from -0.5 mg to $+0.3$ mg from the first weight.

After each experiment the precipitate was brushed out of the crucible and the crucible weighed. Since the crucibles used for igniting the alumina were cleaned between the experiments the individual weights are not given, but only the difference between the weighing before and after the experiment. These differences vary from $+0.6$ mg to -0.6 mg. The control crucible was cleaned before experiments 7, 9, 11, and 13. The weights and calculations given in Table 4 are all based upon the weight of the crucible at the beginning of the experiment. The losses in weight from the initial weighing of the ignited alumina for the final weighing in only a very few cases amount to as much as 1 mg and in no case to as much as 2 mg. These experiments, as the experiments with silica, indicate that in weighing a platinum crucible under conditions of analysis,

TABLE 4.—*Results of determinations of alumina in ammonia alum.*

Experiment No.	Change in weight of crucible during experiment.	Weight of control crucible at beginning of experiment.	Weight of control crucible at end of experiment.	Change in weight of control crucible during experiment.	Weight of ammonia alum taken.	Alumina after 3 hours over burner.		Alumina after 4 hours over burner.		Alumina after 15 minutes blasting following 4 hours over burner.		Alumina after slow ignition (about 45 minutes) and 15 minutes blasting.		Alumina after slow ignition (about 45 minutes) and 25 minutes blasting.		Correction.	
	Mg.	*Grams.*	*Grams.*	*Mg.*	*Grams.*	*Mg.*	*Per ct.*	*Mg.*	*Per ct.*	*Mg.*	*Per cent.*	*Mg.*	*Per cent.*	*Mg.*	*Per cent.*	*Mg.*	*Per cent.*
1	(1)	16.0469	16.0469	0.0	1.3735	152.1	11.08	152.4	11.10	151.4	11.02	.....	.....	.....	.....	(1)	(1)
2	(1)				1.1831	133.3	11.27	132.9	11.23	132.7	11.22	.....	.....	.....	.....	(1)	(1)
3	−0.3	16.0468	16.0468	.0	1.6159	183.4	11.35	183.4	11.35	181.6	11.24	.....	.....	.....	.....	0.5	0.03
4	− .3				1.4555	164.7	11.32	163.5	11.23	162.9	11.19	.....	.....	.....	.....	(1)	(1)
5	+ .1	16.0468	16.0470	+ .2	1.3985	160.6	11.48	160.3	11.46	159.5	11.41	.....	.....	.....	.....	.0	.00
6	− .2				1.0286	114.8	11.16	114.4	11.12	114.5	11.13	.....	.....	.....	.....	1.8	.17
7	− .6	16.0464	16.0461	− .3	1.2434	140.3	11.28	140.1	11.27	140.0	11.26	.....	.....	.....	.....	.2	.02
8	− .3				1.0581	118.5	11.20	118.0	11.15	117.8	11.13	.....	.....	.....	.....	.7	.07
9	− .4	16.0444	16.0439	− .5	1.8721	210.1	11.22	209.7	11.20	208.7	11.15	.....	.....	.....	.....	1.1	.06
10	− .5				1.5296	172.5	11.28	171.9	11.24	171.4	11.21	.....	.....	.....	.....	.6	.04
11	+ .6	16.0425	16.0426	+ .1	1.5260	.....	.....	.....	.....	.....	.....	174.5	11.44	173.7	11.39	.0	.00
12	− .4				1.2408	.....	.....	.....	.....	.....	.....	139.6	11.25	139.5	11.24	.2	.02
13	.0	16.0415	16.0418	+ .3	1.3264	152.1	11.47	152.1	11.47	151.5	11.42	.....	.....	.....	.....	(1)	(1)
14	+ .5				1.2361	139.3	11.27	139.3	11.27	139.0	11.25	.....	.....	.....	.....	.9	.07
15	+ .3	16.0419	16.0419	.0	1.4354	165.2	11.51	164.7	11.47	163.9	11.42	.....	.....	.....	.....	(1)	(1)
16	− .2				1.3556	154.2	11.37	154.2	11.37	153.8	11.35	.....	.....	.....	.....	(1)	(1)

[1] Not determined.

the error in weight may amount to as much as a milligram if not more. It therefore appears that heating alumina in the manner indicated for three or four hours over a burner reduced it to a con stant weight within the limit of accuracy of such a determination.

All weights throughout this whole series of experiments were made using an accurately standardized set of weights. The weights of 1 gram and upward were lacquered brass and the fractional weights were platinum. An error of as much as a milligram made in weighing a 15 cc platinum crucible is apparently due to condensation on the surface of either crucible or the weights or both. It seems to be illogical to assume that all these errors are due to condensation on the surface of a platinum crucible and not on the lacquered brass weights. The writers have observed the same apparent difference in the weight of a platinum crucible which was kept in a balance case under exactly the same conditions as the weights, and it is certainly reasonable to suppose that a piece of bright platinum would be less subject to variations in condensation on its surface than lacquered articles of like weights.

It is the custom in some works' laboratories to brush precipitates such as silica from the crucible on to a balanced watch glass for weighing, thus saving the time used in weighing the crucible. The method of weighing the crucible and then weighing the crucible with the precipitate is generally considered more accurate and is, of course, necessary for such substances as lime, but it is very doubtful whether this method of weighing is really as accurate as the method of transferring the precipitate to a balanced watch glass, where the weight would be much smaller and the error would be only that due to error in weighing the precipitate itself, and not the relatively large error which is due to weighing a heavy crucible. An assayer would not think of weighing his annealing cup with the gold in it and then weighing the empty annealing cup and getting the gold by difference, and it is probable that, for substances like alumina and silica, weighing after the manner of the assayer would be conducive to more accurate results than by following the regular method.

This is aside, however, from the object of this paper. An attempt has been made to determine whether alumina and silica can be ignited to constant weight without the use of a blast lamp, and as a result the authors are of the opinion that with slow initial ignition and final heating at the highest temperature that can be obtained with a burner, silica is practically always reduced to constant weight in two hours and generally in one hour. Alumina requires a somewhat longer period. About three hours reduces the weight to within the limit of error of the determination.

Approved:

JAMES WILSON,
Secretary of Agriculture.

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